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Ajay K. Misra  
*Sverdrup Technology, Inc.*  
*Lewis Research Center Group*  
*Brook Park, Ohio*

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# INTERFACE CONSIDERATIONS IN $\text{Al}_2\text{O}_3/\text{NiAl}$ COMPOSITE

Ajay K. Misra  
Sverdrup Technology, Inc.  
Lewis Research Center Group  
Brook Park, OH 44142

## ABSTRACT

The fiber-matrix interface requirements in an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite were examined from theoretical considerations. Several factors that influence the interface bonding requirements were analyzed. These include: (1) residual stresses due to fiber-matrix CTE mismatch, (2) matrix cracking stress at room temperature, (3) fracture toughness at room temperature, (4) load transfer from the matrix to the fiber and ultimate tensile strength at the use temperature, and (5) creep resistance at high temperature. A relatively weak fiber-matrix bond, with an interfacial shear strength of ~15-20 MPa, might be sufficient for attaining the desired mechanical properties in the fiber direction at the use temperature. A weak fiber-matrix bond is also beneficial for increasing the fracture toughness of the composite at room temperature. In contrast, a strong fiber-matrix bond is required to withstand some of the residual stresses resulting from the fiber-matrix CTE mismatch, which are not likely to be reduced significantly by interface coatings. A relatively strong bond is also beneficial in increasing the matrix cracking stress at room temperature. Various interface coating options to accommodate the conflicting bonding requirements were reviewed. One viable coating option is to incorporate a thick, ductile interface layer well bonded to both the fiber and the matrix.

## INTRODUCTION

Intermetallic compounds are increasingly becoming important as high temperature structural materials for future aerospace engine applications. One intermetallic compound, NiAl, is attractive because of its high melting point, low density, and excellent oxidation resistance. Two severe limitations to the use of NiAl as a high temperature structural material are low fracture toughness (or lack of ductility) at room temperature and poor creep resistance plus low strength at high temperature. Reinforcement of NiAl with continuous, strong fibers is currently being explored to circumvent these two limitations. Single crystal (c-axis)  $\text{Al}_2\text{O}_3$  fiber is attractive as a potential reinforcement for NiAl because of its high modulus, high temperature strength, and chemical compatibility with the matrix. However, the coefficient of thermal expansion (CTE) of  $\text{Al}_2\text{O}_3$  ( $8.9 \times 10^{-6} \text{ K}^{-1}$ ) is lower than that of NiAl ( $\sim 15\text{-}16 \times 10^{-6} \text{ K}^{-1}$ ), which will result in residual stresses in the composite under thermal cycling conditions.

The fiber-matrix interface has a strong influence on the mechanical properties of any composite, and  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites will be no exception. The interface design becomes particularly difficult for NiAl-based composites because NiAl is brittle at lower temperatures and ductile at higher temperatures, with a ductile-to-brittle-transition temperature (DBTT) of  $\sim 500 \text{ K}$  (ref. 4). The fibers in a NiAl-based composite are expected to perform a multitude of functions which include: (1) carrying the load at high temperature, (2) increasing creep resistance of the matrix, and (3) increasing the fracture toughness at room temperature. There have been a number of studies (refs. 1-3) dealing with the effect of interfaces<sup>1</sup> on the mechanical properties of brittle matrix composites.

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<sup>1</sup> The term "interface" refers to the fiber-matrix interface

It is generally agreed that a weak fiber-matrix interface increases the fracture toughness of the composite by allowing debonding at the interface ahead of the advancing crack tip and subsequent fiber pull-out. A relatively "strong" bond might be required for effective load transfer between the matrix and the fiber at high temperature and to prevent the matrix from creeping at high temperature, although the optimum bond strength required for desired high temperature properties is not yet known. Some recent studies (refs. 5 and 6) on metal matrix composites indicate that some degree of weakness at the interface might be desirable for attaining optimum mechanical properties at the use temperature. The difficulty in designing an optimum interface is further compounded by the fiber-matrix CTE mismatch, which might impose additional restrictions on the interface bonding requirements.

The objective of this paper is to analyze the interface requirements in an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite<sup>2</sup> and to identify the optimum interface characteristics for this composite. The factors considered to define the interface requirements include: (1) fiber-matrix CTE mismatch and the resulting residual stresses, (2) matrix cracking stress at room temperature, (3) fracture toughness at room temperature, and (4) high temperature strength and creep resistance. It is hoped that once the optimum interface requirements are determined, suitable interface coatings can be identified.

### **EFFECT OF FIBER-MATRIX CTE MISMATCH ON BONDING REQUIREMENTS**

The average CTE of NiAl in the temperature range of 300 to 1300 K is about  $15 \times 10^{-6} \text{ K}^{-1}$  (ref. 7), as compared to about  $9 \times 10^{-6} \text{ K}^{-1}$  for c-axis  $\text{Al}_2\text{O}_3$  (ref. 8). The fiber-

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<sup>2</sup>The fiber volume fraction and diameter in this study were assumed to be 0.3 and 125  $\mu\text{m}$ , respectively.

matrix CTE mismatch will result in residual stresses in the composite under thermal cycling conditions. Of the three stress components, radial( $\sigma_r$ ), hoop( $\sigma_\theta$ ), and axial( $\sigma_z$ ), only the radial stress at the fiber-matrix interface (which is the stress normal to the interface) has a direct effect on the integrity of the fiber-matrix bond. A compressive radial stress provides a clamping action on the fiber, and is thus beneficial to the integrity of the fiber-matrix bond. On the other hand, a radial tensile stress can cause debonding of the fiber from the matrix for a weak fiber-matrix bond. In addition, the CTE mismatch-induced residual stresses can result in interface shear near the fiber ends, which can also affect the integrity of the fiber-matrix bond.

Effect of thermal cycling on residual radial stresses at the interface: A concentric cylinder model (refs. 9 and 10) was used to calculate the residual stresses at the interface. Residual stresses, if large enough, can result in plastic deformation of the matrix, thereby relieving the residual stresses. Von-Mises criteria (ref. 11) was used to examine if residual stresses can cause matrix yielding. According to this criteria, the matrix will yield if the effective stress,  $\sigma_{eff}$ , defined as

$$\sigma_{eff} = \frac{\sqrt{(\sigma_r - \sigma_\theta)^2 + (\sigma_\theta - \sigma_z)^2 + (\sigma_z - \sigma_r)^2}}{\sqrt{2}} \quad (1)$$

becomes greater than the uniaxial yield stress ( $\sigma_y$ ) of the matrix. The various matrix and fiber properties used in the calculations are given in Table I. Only one thermal cycle, consisting of cooling of the composite from high temperature to room temperature and subsequent heating from room temperature to elevated temperature, was considered in this study.

Consider the cooling cycle first, which may correspond to cooling from the

processing temperature to room temperature. A stress-free temperature of 1100 K (~ one-half the melting point of NiAl) was assumed in the calculations. The individual stress components in the matrix at the fiber-matrix interface during cooling are compressive for radial, and tensile for both hoop and axial directions. The magnitudes of these stresses in the matrix at the fiber-matrix interface after cooling from 1100 K to room temperature, assuming elastic behavior for both the fiber and the matrix, are -442 MPa for  $\sigma_r$ , 821 MPa for  $\sigma_\theta$ , and 541 MPa for  $\sigma_z$ . The magnitude of these stresses would be lower (less compressive) because of plastic deformation of the matrix during cooling, as explained below.

Examination of the residual  $\sigma_{\text{eff}}$  in the matrix at the fiber-matrix interface during cooling (figure 1) shows that except for the first 80 K of cooling from the assumed stress-free temperature of 1100 K, the  $\sigma_{\text{eff}}$  is greater than the yield stress of matrix. Thus, the residual stresses during cooling will be relieved by plastic deformation of the matrix. From figure 1 the slope of the  $\sigma_{\text{eff}} - T$  (T is temperature) curve is more negative than that of the  $\sigma_y - T$  curve at all temperatures within the temperature range shown in this figure. This means that as temperature decreases, the CTE mismatch-induced  $\sigma_{\text{eff}}$  in the matrix increases at a faster rate than the matrix yield stress. Therefore, once the temperature is reached where  $\sigma_{\text{eff}}$  becomes equal to  $\sigma_y$ , the matrix will yield and continue to deform plastically throughout the cooling period, thereby relieving the residual stresses.

Next, consider the heating cycle. Residual stresses in the matrix during subsequent heating from room temperature are a function of the residual stresses at the end of the previous cooldown period. The residual  $\sigma_{\text{eff}}$  at the beginning of the heating cycle was assumed to be  $\sigma_y$  for the matrix at room temperature -- which would be true for an ideal

plastic matrix. Residual stresses,  $\sigma_r$ ,  $\sigma_\theta$ , and  $\sigma_z$ , during heating were first calculated using the concentric cylinder model (with the assumption of elastic behavior), assuming zero residual stresses at the beginning. A  $\sigma_{eff}$  value was calculated from these individual stress components, and then subtracted from the  $\sigma_{eff}$  at the beginning of the heating cycle to obtain  $\sigma_{eff}$  at any given temperature during heating.

Calculated  $\sigma_{eff}$  in the matrix at the fiber-matrix interface during heating is shown in figure 2 as a function of temperature, along with the tensile and compressive yield stress (the magnitudes of both are assumed to be the same) of NiAl. The  $\sigma_{eff}$  is tensile at the beginning of the heating cycle, decreases with increase in temperature, becomes zero at  $\sim 490$  K, and then becomes compressive, the magnitude of the compressive stress increasing with increasing temperature. Plastic deformation of the matrix during heating will occur when the magnitude of the compressive  $\sigma_{eff}$  becomes greater than the compressive yield stress of the matrix; this corresponds to temperatures greater than  $\sim 620$  K in figure 2. The magnitude of the compressive  $\sigma_{eff}$  will be maximum at 580 K, after which it will decrease (become less negative) due to stresses being relieved by plastic deformation of the matrix.

There are two important differences between the stress states during cooling and heating cycles. First, during cooling, the matrix starts yielding within the first few degrees from the onset of cooling and will continue to deform plastically throughout the cooling cycle. However, during the subsequent heating cycle, the matrix undergoes elastic deformation for a significant portion of the heating cycle. Second, the effective stress throughout the cooling period is tensile, but becomes compressive during the subsequent heating cycle after becoming zero at an intermediate temperature.



The abovementioned differences between the stress states during cooling and heating cycles have an important bearing on the radial stress at the interface. This is schematically shown in figure 3. The radial stress is compressive during the cooling cycle. During subsequent heating, the radial stress becomes less compressive with increase in temperature (i.e., compressive stress at the interface is released as temperature increases), finally becoming zero at the temperature where  $\sigma_{\text{eff}}$  is zero (~490 K in figure 2), which may be called the stress-free temperature during heating. Heating beyond this stress-free temperature results in a radial tensile stress at the interface, which is in contrast to the radial compressive stress developed during cooling.

Because of the compressive radial stress at the interface during cooling, the interface is subjected to a clamping pressure, which helps maintain the integrity of the fiber-matrix bond, even if the bonding is due to only frictional effects at the interface (i.e., no chemical bond). However, for a frictionally-bonded interface, the bonding will be lost when the radial stress becomes tensile during the heating cycle. Therefore, a chemical bond between the fiber and the matrix is required for an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite, and the bond must have sufficient strength to withstand the radial tensile stress during heating. The magnitude of the residual  $\sigma_r$  at the interface for heating from 490 to 620 K -- the temperature range in which the matrix undergoes elastic deformation due to the residual stresses -- is about 75 MPa, which is also likely to be the maximum value for  $\sigma_r$  during heating because of the stresses being relieved by plastic deformation of the matrix beyond 620 K. The magnitude of the maximum radial tensile stress during heating will probably depend on the time-dependent viscoplastic properties of the matrix and might be different than 75 MPa. However, the conclusion that the interface will be subjected to

radial tensile stresses during the heating cycle is not likely to change even if time-dependent properties of the matrix are taken into account.

The requirement of a strong fiber-matrix bond for an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite arises due to matrix yielding during the cooldown cycle. Had the residual stresses during cooling been elastic all the way from the stress-free temperature to room temperature, no radial tensile stresses would have developed at the fiber-matrix interface during heating; radial compressive stresses developed during cooling would simply have been gradually lowered during heating, ultimately becoming zero.

**Interface shear:** Because the CTE of the matrix is greater than that of the fiber, the matrix shrinks more than the fiber during cooling. This results in a stressed interface with the fiber in axial compression and the matrix in axial tension. The interface is subjected to shear because the load which develops the axial compressive stress must be transmitted by shear stresses at the interface. During the initial stages of cooling, the interface is stressed elastically by shear, the magnitude of the shear stress increasing with decrease in temperature. However, the interfacial shear stress cannot exceed the shear yield stress of the matrix ( $\tau_{\text{yield}}$ ). Thus, the  $\tau_{\text{yield}}$  sets an upper limit for the interface shear stress. The interface shear strength of the fiber-matrix bond must be greater than the shear stresses developed during cooling; otherwise, debonding at the interface can be expected.

In the elastic regime, the interfacial shear stress during cooling can be calculated from the shear lag model (ref. 14). The interfacial shear stress  $\tau(z)$  is

$$\tau(z) = \frac{E_f d \theta \beta}{4 \cosh(\beta L/2)} \sinh(\beta z) \quad (2)$$

where

$$\beta = \sqrt{\frac{8 G_m}{E_f d^2 \ln(v_f^{-1/2})}} \quad (3)$$

and  $E_f$  is the elastic modulus of the fiber,  $L$  the fiber length,  $d$  the fiber diameter,  $z$  the distance from the fiber center along the longitudinal axis,  $v_f$  the fiber volume fraction, and  $G_m$  the shear modulus of the matrix. For continuous fiber reinforced composites, the interface shear is maximum near the fiber ends. From eqns. [2] and [3] the maximum interface shear stress<sup>3</sup> for each 100 K of cooling is ~122 MPa. However, because of the low  $\tau_{\text{yield}}$  of the NiAl matrix ( $\tau_{\text{yield}}$  assumed to be one-half of the yield stress in uniaxial direction), as shown in Table II, the matrix will yield before the shear stress at the interface reaches such high values. Thus, the  $\tau_{\text{yield}}$  values shown in Table II are the limiting values for the interface shear stress at any given temperature during cooling, and the shear strength of the fiber-matrix bond at any given temperature must be greater than the  $\tau_{\text{yield}}$  for the matrix.

Whether a frictional bond can provide the required interface shear strength at high temperature is questionable. For a frictional bond, the interface shear strength ( $\tau$ ) is

$$\tau = \sigma_f \cdot \mu \quad (4)$$

where  $\mu$  is the coefficient of friction at the fiber-matrix interface. Take, for example, cooling

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<sup>3</sup>For simplicity, the maximum interface shear stress was calculated with the assumption of temperature-independent elastic modulus and CTE values for both the fiber and the matrix.

of the composite from the assumed stress-free temperature of 1100 K to 1000 K. Radial compressive stress at the interface after cooling to 1000 K, calculated using concentric cylinder model with the assumption of elastic behavior for all constituents, is ~50 MPa. Assuming a  $\mu$  of 0.3 -- typical for metal-ceramic interfaces -- the interface shear strength for a  $\sigma_r$  of 50 MPa is 15 MPa, which is much lower than the required interface shear strength of 51 MPa at 1000 K ( $\tau_{\text{yield}}$  of matrix at 1000 K). The  $\sigma_r$  at 1000 K will actually be lower than 50 MPa because of stresses being relieved by matrix yielding. Thus, the interface shear strength due to frictional effects only will be even lower than 15 MPa. Clearly, a frictionally-bonded interface will not have the required interface shear strength at 1000 K. The same conclusion is true at other temperatures throughout the cooling period. Thus, a chemical fiber-matrix bond with adequate interface shear strength at high temperatures is required.

### **MATRIX CRACKING STRESS AT ROOM TEMPERATURE**

Because of the lack of ductility in NiAl at room temperature, NiAl-based composites at room temperature can be considered to be brittle matrix composites, similar to the ceramic matrix composites. In a brittle matrix composite, the matrix cracking stress may be substantially greater than the fracture stress (or the ultimate tensile stress) of the unreinforced matrix (ref. 15). The definition of the matrix cracking stress ( $\sigma_{cr}$ ) is shown in figure 4, which is a typical stress-strain curve for a brittle matrix composite. The initial portion of the stress-strain curve follows the rule of mixture based on the matrix and fiber moduli. Extensive matrix cracking occurs at A; the stress at point A is the matrix cracking stress.

It is desirable to have a high enough  $\sigma_{cr}$  value such that the matrix does not crack

due to residual tensile stresses. Furthermore, a high  $\sigma_{cr}$  will enable the composite to sustain a higher load at room temperature without matrix cracking. The  $\sigma_{cr}$  is a strong function of the interface shear strength and the residual axial tensile stress in the matrix (ref. 15). The matrix cracking model of Budiansky, Hutchinson, and Evans - to be referred as the BHE model -- (ref. 15) considered only frictional shear stress at the interface. Recently, Sutcu and Hillig (ref. 16) have extended the BHE model to include the combined effects of friction and adhesion (or bonding) at the fiber-matrix interface on the matrix cracking stress. The Sutcu and Hillig model (ref. 16) will be used here to derive the steady state matrix cracking stress at room temperature for an  $Al_2O_3/NiAl$  composite.

The Sutcu and Hillig model assumes that debonding at the fiber-matrix interface adjacent to the matrix crack consumes a material's specific debond energy  $\Gamma_d$  and that frictional sliding along the debonded length is resisted by a constant interfacial frictional shear stress  $\tau_f$ . The steady state  $\sigma_{cr}$  is

$$\frac{\sigma_{cr} + E_f \sigma_z / E_m}{\sigma_o} = [X + \sqrt{X^2 + Y^{18}/729}]^{1/3} + [X - \sqrt{X^2 + Y^{18}/729}]^{1/3} \quad (5)$$

where

$$X = Y^9/54 + Y^3/2 + 4 \left( \frac{V_f \Gamma_d}{\rho V_m \Gamma_m} \right)^{3/2} \quad (6)$$

$$Y = \left( \frac{9R \tau_f^2 V_f v}{G_m V_m \Gamma_m \rho} \right)^{1/6} \quad (7)$$

The various terms in eqns. [5] - [11] are defined as follows:  $V_f$  and  $V_m$ , volume fractions of fiber and matrix, respectively;  $E_f$  and  $E_m$ , elastic modulus of fiber and matrix,

$$\sigma_o = \left[ \frac{V_f E_f E \Gamma_m \rho}{R E_m} \right]^{1/2} \quad (8)$$

$$\sigma_1 = \left[ \frac{6 V_f^2 E_f E^2 \tau_f \Gamma_m}{V_m E_m^2 R} \right]^{1/3} \quad (9)$$

$$\rho^2 = \frac{4 E G_m}{V_m E_m E_f \nu} \quad (10)$$

$$\nu = \frac{-2 \ln V_f - V_m (3 - V_f)}{2 V_m^2} \quad (11)$$

respectively;  $E$ , elastic modulus of the composite  $= V_f E_f + V_m E_m$ ;  $R$ , the fiber radius;  $\tau_f$ , the interface frictional shear strength;  $G_m$ , shear modulus of the matrix,  $= E_m / 2(1 + \nu_m)$ , where  $\nu_m$  is the Poisson's ratio for the matrix;  $\Gamma_m$ , fracture energy of the matrix;  $\Gamma_d$ , interface debond energy or fracture energy of the interface.

The fracture energy  $\Gamma_m$  of NiAl was calculated from the expression (ref. 17)

$$\Gamma_m = \frac{K_{IC}^2}{E_m} \quad (12)$$

where  $K_{IC}$  is the fracture toughness of the matrix. Using a fracture toughness of 5 MPa $\sqrt{m}$  for NiAl (ref. 18),  $\Gamma_m$  is 141 J/m<sup>2</sup> (1.41\*10<sup>-4</sup> MPa.m). A  $\sigma_z^m$  value of 320 MPa, which is the average  $\sigma_z^m$  obtained from the finite-element calculations by Arnold (ref. 19), was used to determine  $\sigma_{cr}$ .

Figure 5 shows the  $\sigma_{cr}$  for an Al<sub>2</sub>O<sub>3</sub>/NiAl composite at room temperature as a function of the interface frictional shear strength for different  $\Gamma_d$  values. A negative  $\sigma_{cr}$  means that matrix can crack without the application of any external stress, i.e., matrix

cracking is due the tensile longitudinal residual stresses only. For a frictionally bonded interface ( $\Gamma_d = 0 \text{ J/m}^2$ ), residual tensile stresses can cause matrix cracking if the  $\tau_f$  is less than  $\sim 60 \text{ MPa}$ . The interface shear strengths for frictionally-bonded  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites have been measured to be in the range of 50 to 150 MPa by Bowman (ref. 20) and  $87 \pm 37 \text{ MPa}$  by Helmann et al. (ref. 21). These high values for the interface shear strength probably explains why no matrix cracking has been observed (ref. 20) in frictionally bonded  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites after cooling from the processing temperature to room temperature.

The  $\sigma_{cr}$  increases with increasing interfacial frictional shear strength. For a given  $\tau_f$  the  $\sigma_{cr}$  increases with increasing  $\Gamma_d$ ; or in other words, the  $\sigma_{cr}$  increases with increasing fiber-matrix bond strength. However,  $\sigma_{cr}$  cannot increase indefinitely with increasing  $\Gamma_d$ ; it reaches a limiting value  $\sigma_o$  corresponding to the no-slip condition at the fiber-matrix interface. The limiting  $\sigma_{cr}$  corresponding to the no-slip condition, calculated from eqn. [8], is  $\sim 359 \text{ MPa}$  for an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite.

A high  $\sigma_{cr}$  is desirable for attaining high strength in the composite at room temperature, and this can be achieved by having a strong fiber-matrix bond with a high  $\Gamma_d$  value. However, it should be noted that the calculated matrix cracking stress is for steady state matrix cracking condition or for situations where multiple matrix cracking occurs. One drawback for a brittle matrix composite with a strong fiber-matrix bond is that the mechanical properties of the composite are dependent on the flaw size, which can result in catastrophic failure of the composite (due to propagation of a single crack). Thus, steady state matrix cracking conditions may never be achieved in a brittle matrix composite with a strong fiber-matrix bond. There probably exists an optimum  $\Gamma_d$  below

which steady state matrix cracking conditions can be obtained.

### FRACTURE TOUGHNESS AT ROOM TEMPERATURE

In brittle matrix composites that fracture by the growth of a single dominant flaw, increasing the fracture toughness of the composite requires debonding at the interface ahead of the advancing crack tip and subsequent fiber pull-out (refs. 1-3).

Contributions due to debonding at the interface: The increase in fracture energy ( $\Delta G_c$ ) due to debonding at the fiber-matrix interface is (ref. 22)

$$\Delta G_c = \frac{V_f L_d S^2}{E} + \frac{4\Gamma_d V_f (L_d/R)}{1-V_f} - V_f L_d E e_T^2 \quad (13)$$

where  $L_d$  is the debond length,  $e_T$  is the misfit strain due to fiber-matrix CTE mismatch, and  $S$  is the fiber strength. The debond length, from Sutcu and Hillig's model (ref. 16), is defined as

$$\rho \frac{L_d}{R} = 3 \left( \frac{\sigma_o}{\sigma_1} \right)^3 \left[ \frac{\sigma_{\sigma} + \sigma_z^m E/E_m}{\sigma_o} - 2 \left( \frac{V_f \Gamma_d}{\rho V_m \Gamma_m} \right)^{1/2} \right] - 1 \quad (14)$$

The debond length and increase in  $\Delta G_c$  were calculated with the following values for different parameters:  $\tau_f = 87$  MPa (ref. 21);  $\Gamma_m = 141$  J/m<sup>2</sup>; fiber strength  $S = 2067$  MPa (300 ksi). Figure 6 gives the debond length and  $\Delta G_c$  due to debonding as a function of the interface debond energy ( $\Gamma_d$ ) for an Al<sub>2</sub>O<sub>3</sub>/NiAl composite. The  $\Delta G_c$  increases with decreasing  $\Gamma_d$ , and considerable increases in the fracture energy (an order of magnitude higher than the fracture energy of unreinforced NiAl) can be obtained only for  $\Gamma_d < 10$  J/m<sup>2</sup>.

Any increase in fracture energy due to debonding at the fiber-matrix interface is



based on the assumption that debonding will occur ahead of the crack tip instead of fiber fracture. It has been suggested (ref. 2) that the fracture or debond energy of the interface ( $\Gamma_d$ ) should be less than 1/4 of the fiber fracture energy ( $\Gamma_f$ ) for debonding to occur at the interface. Based on a fracture energy of 20 J/m<sup>2</sup> for single crystal Al<sub>2</sub>O<sub>3</sub> (ref. 23), a  $\Gamma_d$  of  $\leq 5$  J/m<sup>2</sup> is required for debonding at the Al<sub>2</sub>O<sub>3</sub>/NiAl interface.

Contributions due to fiber pull-out: The contribution to fracture toughness from fiber pull-out is due to work done against frictional forces in extracting broken fibers from matrix crack faces. The increase in fracture energy ( $\Delta G_c$ ) due to fiber pull-out is a function of the critical load transfer length ( $l_c$ ), and is maximum when the embedded fiber length ( $l$ ) equals  $l_c$  (ref. 24). The maximum increase in fracture energy (ref. 24) due to fiber pull-out is

$$\Delta G_c^{max}(pull-out) = \frac{V_f S l_c}{12} \quad (15)$$

where  $l_c = S \cdot R / \tau_f$ . The maximum  $\Delta G_c$  due to fiber pull-out for  $S = 2067$  MPa (300 ksi of fiber strength) and for a  $\tau_f$  of 87 MPa is 77 kJ/m<sup>2</sup>, which is significantly higher than the fracture energy of unreinforced NiAl ( $\sim 141$  J/m<sup>2</sup>).

The actual increase in  $\Delta G_c$  due to fiber pull-out is likely to be lower than 77 kJ/m<sup>2</sup> because the fiber pull-out length ( $l$ ) is likely to be less than  $l_c$ . For  $l > l_c$ , the fraction of fibers pulled out is  $l_c/l$  (based on normal probability), and the corresponding increase in fracture energy is (ref. 24)

$$\Delta G_c(pull-out) = \frac{V_f S l_c}{12} \left( \frac{l_c}{l} \right) \quad (16)$$

If  $l_c/l$  is 0.1 (which means that 10 % of the fibers are pulled out), the  $\Delta G_c$  is 7.7 kJ/m<sup>2</sup>,

which is still substantial in comparison to the fracture energy of unreinforced NiAl. Clearly, fiber pull-out can contribute substantially to the fracture toughness of an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite.

The  $\Delta G_c$  due to fiber pull-out can be increased by increasing the fiber strength and by decreasing the interface frictional shear stress. Typical  $\tau_f$  values for ceramic matrix composites exhibiting significant fiber pull-out are in the range of 2 to 40 MPa (ref. 3). The  $\tau_f$  in frictionally bonded  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites is in the range of 50 to 100 MPa (refs. 20 and 21). Even with such high  $\tau_f$  values, significant fiber pull-out has been observed for frictionally bonded  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites (ref. 20). Thus, fiber pull-out can contribute significantly toward increasing the fracture toughness of the composite at room temperature, provided the interface debonding criterion is satisfied, i.e.,  $\Gamma_d \leq 5 \text{ J/m}^2$ .

#### **LOAD TRANSFER AT HIGH TEMPERATURE**

The applied stress is shared between the matrix and the fibers, the load being transferred to the latter by shear at the fiber-matrix interface. Because of very low yield stress of NiAl at high temperatures, the matrix will yield under a very small load and, therefore, the interface shear stress cannot exceed the shear yield stress of the matrix. Thus, for effective load transfer from the matrix to the fiber, the interface shear strength must be greater than the shear yield stress of the matrix; otherwise debonding will occur at the fiber-matrix interface before the onset of matrix yielding. Consider a use temperature of 1300 K. Based on an uniaxial yield stress of  $\sim 40 \text{ MPa}$  at 1300 K (ref. 4), the shear yield stress, assumed to be one-half of the yield stress, is 20 MPa. Thus, the interfacial shear strength for an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite must be greater than 20 MPa for effective load transfer from the matrix to the fiber. Because  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites are

likely to be used at temperatures greater than 1373 K, the interface shear strength requirements will even be lower.

Although the required interface shear strength for effective load transfer at high temperature is relatively low, it is still questionable whether a purely frictional bond can meet the requirements. This is due to lack of any compressive radial stress (or the clamping pressure) at high temperature. Thus, some sort of chemical bond at the fiber-matrix interface might be required, although the bond strength does not have to be nearly as high as that required to withstand radial tension during heating.

### **ULTIMATE TENSILE STRENGTH AT HIGH TEMPERATURE**

At high temperature fibers carry most of the load. With increasing tensile loading, the weakest fiber will fracture first. The broken fiber will then cause high stress concentration in adjacent fibers, thus causing rapid breakage of all the fibers and the resulting low fracture strain for the composite. Debonding at the interface near the fiber cracks can reduce stress concentration in the adjacent fibers, thereby increasing the fracture strain of the composite. Debonding at the interface region near the fiber cracks will also increase the fracture toughness of the composite by increasing the contribution due to plastic deformation of the matrix (ref. 5) . Debonding at the fiber-matrix interface requires an interface with a relatively low strength, i.e., an interface with a  $\Gamma_i \leq 5 \text{ J/m}^2$ . However, as discussed before, the interface shear strength must be greater than the shear yield stress of the matrix for effective load transfer from the matrix to the fiber. An optimum interface bond which will allow for effective load transfer as well as debonding near the fiber cracks is probably one with a shear strength slightly higher than the shear yield stress of the matrix.

## **CREEP RESISTANCE AT HIGH TEMPERATURE**

It has been the general belief that a strong fiber-matrix bond is required for improving the creep resistance of the composite at high temperature. However, some of the recent theoretical analyses (refs. 25 and 26) have shown this to be the case only for discontinuous fiber reinforced metal matrix composites. For continuous fiber reinforced composites, the creep rate is not a function of the interface strength (ref. 25), provided the interface has sufficient shear strength to allow for effective load transfer from the matrix to the fiber. For an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite this means that the interface shear strength must be greater than the shear yield stress of NiAl at the use temperature for creep rate to be independent of the interface bond strength.

## **SUMMARY OF INTERFACE BOND REQUIREMENT**

The various bonding requirements due to different factors are summarized in Table III. A relatively weak bond with an interfacial shear strength of 10 to 20 MPa may be sufficient for attaining optimum high temperature mechanical properties of the composite in the fiber direction. Such a weak bond will also improve the fracture toughness of the composite at room temperature by allowing for debonding at the interface and subsequent fiber pull-out. In contrast, a relatively strong bond, with transverse strength on the order of 70 MPa and with an interfacial shear strength of 50 to 100 MPa at high temperature (600 to 1000 K), is required to withstand radial tension during heating and to withstand interface shear near the fiber ends during cooling, both of which are caused by residual stresses due to fiber-matrix CTE mismatch. Thus, the requirement of a strong fiber-matrix bond can be eliminated by lowering the CTE mismatch induced residual stresses. Because the development of radial tensile stresses at the interface during

heating is a result of matrix yielding during the cooling cycle, it is desirable that the residual stresses be lowered to levels where matrix yielding cannot occur.

### **EFFECT OF INTERFACE COATINGS ON RESIDUAL STRESS**

One potential means by which residual stresses can be lowered is to apply an interface coating that can compensate for the fiber-matrix CTE mismatch. Arnold et al. (ref. 10) have employed concentric cylinder finite element models to determine the optimum characteristics for interface coatings that can reduce the CTE mismatch induced residual stresses in intermetallic matrix composites. One of the important findings from this study is that the CTE of the interface coating should be higher than the CTE of the matrix for reducing the residual stresses in the system. However, there are some practical limitations to using an interface layer with a CTE higher than that of the matrix. Because the CTE of NiAl is already very high, it will be extremely difficult, if not impossible, to find a coating composition with a CTE higher than that of NiAl. There are a few metals like Cu, which have CTEs higher than that of NiAl, but these coatings pose serious chemical compatibility problems. Thus, the use of a coating with a CTE higher than that of NiAl may not be practical for  $\text{Al}_2\text{O}_3/\text{NiAl}$  composites.

The effect of a few practical coating compositions on the residual stresses in the composite were examined using the concentric three cylinder model (ref. 27), assuming elastic behavior for all the constituents. The interface layer compositions examined were  $\text{Y}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{ZrO}_2$ , and  $\text{La}_2\text{O}_3$ . The various properties for these oxides were taken from references 8 and 13.

Figure 7 shows the effect of different interface layers on residual  $\sigma_{\text{eff}}$  in the matrix at the matrix-coating interface during cooling, along with the matrix yield stress.  $\text{Y}_2\text{O}_3$  and

ZrO<sub>2</sub> interfacial coatings have marginal effects on the residual stresses in the matrix. The other two coating compositions considered here, La<sub>2</sub>O<sub>3</sub> and MgO, reduce the matrix  $\sigma_{\text{eff}}$  somewhat compared to that without any coating, but not large enough to prevent matrix yielding. While only a few coating compositions are examined here, and there might be several other coating options, it is probably unlikely that an interface coating can reduce residual stresses in the matrix during cooling to levels where  $\sigma_{\text{eff}}$  is less than  $\sigma_y$ , especially if the coating thickness is to be maintained at a reasonable level.

### INTERFACE COATING OPTIONS

Because the interface coatings are not likely to lower the residual stresses to levels where matrix will not yield, a strong fiber-matrix bond becomes one of the prime requirements for an Al<sub>2</sub>O<sub>3</sub>/NiAl composite, primarily to withstand radial tensile stresses during heating, the magnitude of which can be on the order of 70 MPa. If the interface strength is too low, the question then arises: how to increase the fracture toughness of the composite at room temperature with such a strong fiber-matrix bond? It is unlikely that the interface with a transverse strength of ~75 MPa will have a  $\Gamma_d$  of  $\leq 5 \text{ J/m}^2$ , which is required for debonding at the interface. One way to increase the fracture toughness of the composite is to have a hybrid composite in which some fibers are strongly bonded to the matrix and others weakly bonded. However, this will reduce the load bearing capacity of the composite at the use temperature.

One viable option for increasing the fracture toughness of the composite is to incorporate a ductile interface layer, well bonded to both the fiber and the matrix. This concept is similar to that in which ductile reinforcements are used to increase the toughness of brittle ceramics and intermetallics (refs. 28 and 29). If a ductile interface

coating can be assumed to behave similar to that of ductile reinforcements, then the increase in fracture energy,  $\Delta G_c$ , due to the incorporation of a ductile interface is

$$\Delta G_c = \frac{\sigma_o f t \chi}{2} \quad (17)$$

where  $\sigma_o$ ,  $f$ ,  $t$ , and  $\chi$  are uniaxial yield strength, volume fraction of the ductile layer, thickness of the ductile layer, and a rupture parameter, respectively. The value of  $\chi$  is a function of debonding at the interface and is normally in the range of one to six (refs. 28 and 29), one for a well bonded interface and six for an interface that can undergo extensive debonding.

Figure 8 shows the increase in  $\Delta G_c$  for an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite with a ductile Mo interface coating well bonded to both the fiber and the matrix ( $\chi = 1$ ) as a function of the ductile layer thickness. Note that coating thicknesses on the order of 8 to 10  $\mu\text{m}$  are required for a two-fold increase in the fracture energy of NiAl (141 J/m<sup>2</sup>). The fracture toughness can be further increased by incorporating an interface having a high yield point.

A strong fiber-matrix bond is likely to have adverse effects on the ultimate tensile strength of the composite, due to crack in one fiber causing stress concentration in adjacent fibers. However, for a highly ductile matrix, which is the case for NiAl at the use temperature, energy dissipation due to plastic flow of the matrix can reduce the stress concentration in fibers adjacent to a broken fiber, provided there is enough matrix material around each fiber. Thus, a high ultimate tensile strength at use temperature can be achieved in an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite by using large diameter fibers so that there is a large amount of matrix material around each fiber.

## CONCLUSION

A chemical fiber-matrix bond is required in an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite to withstand residual stresses resulting from fiber-matrix CTE mismatch, to increase the matrix cracking stress at room temperature, to transfer load from the matrix to the fiber at use temperature, and to increase creep resistance of the matrix. A relatively weak bond, with interface shear strengths on the order of 10 to 20 MPa, is sufficient for attaining the desired mechanical properties in the fiber direction at the use temperature. On the other hand, a strong bond having transverse strength on the order of 75 MPa<sup>4</sup> and with an interface shear strength of ~50-100 MPa at high temperatures is required to withstand radial tensile stresses at the interface during heating and to withstand interface shear near the fiber ends, each of which is due to residual stresses because of fiber-matrix CTE mismatch. Because the interface coatings are not likely to lower the residual stresses in the matrix to levels below the matrix yield stress, a strong fiber-matrix bond becomes one of the prime requirements for the  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite. A strong fiber-matrix bond, if properly optimized such that multiple matrix cracking conditions are satisfied, can increase the matrix cracking stress at room temperature. The major disadvantage of having a strong fiber-matrix bond is low fracture toughness at room temperature.

One potential solution for increasing the room temperature fracture toughness of an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite with a strong fiber-matrix bond is to incorporate a thick (probably 8 to 10  $\mu\text{m}$ ), ductile interface coating well bonded to both the fiber and the matrix. The optimum characteristics of this ductile interface requires further study.

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<sup>4</sup>The required interface strength values are based on simplistic models and should not be taken as absolute values.



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**TABLE I**

Properties of NiAl and Al<sub>2</sub>O<sub>3</sub> fiber used in  
residual stress calculations  
(references are given in brackets)

Property	NiAl	Al <sub>2</sub> O <sub>3</sub> fiber
Elastic modulus (GPa)	188.8 - 0.04T (7)	463-0.04T (12)
CTE (x10 <sup>6</sup> K <sup>-1</sup> )	0.143E+02- 0.1013E-02*T + 0.2368E-05*T <sup>2</sup> + 0.1389E-11*T <sup>3</sup> (7)	0.5932E+01 + 0.425E-02*T -0.634E-06*T <sup>2</sup> - 0.7017E-11*T <sup>3</sup> (8)
Poissons ratio	0.32 (7)	0.22 (13)
Yield strength (MPa)	326.4 - 0.223T for T > 600 K (least square fit of ref. 4 data) 315 MPa at 300 K	

**TABLE II**

Shear yield stress ( $\tau_{\text{yield}}$ ) of NiAl  
at different temperatures

$$(\tau_{\text{yield}} = 1/2 \sigma_y)$$

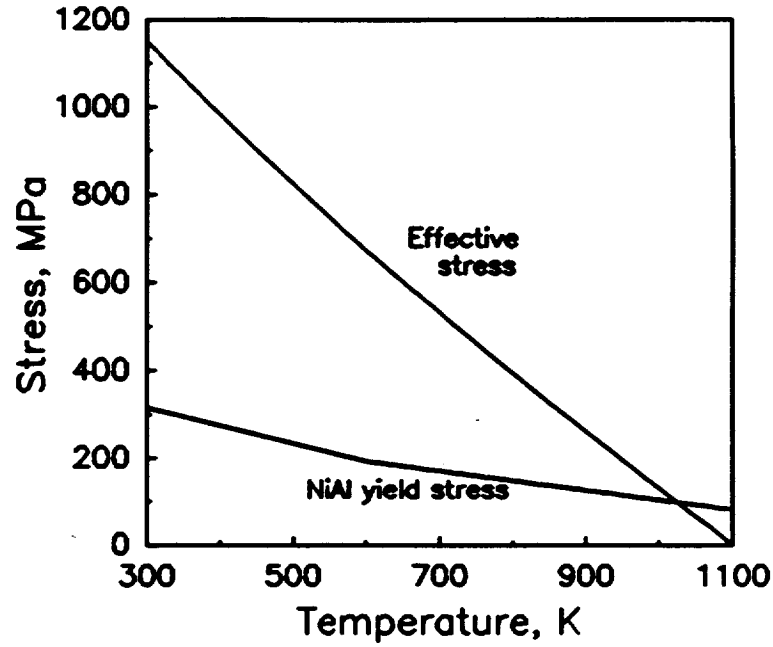
Temperature (K)	$\tau_{\text{yield}}$ (MPa)
1100	40
1000	51
900	63
800	73
600	95

**TABLE III: Summary of interface bonding requirements**

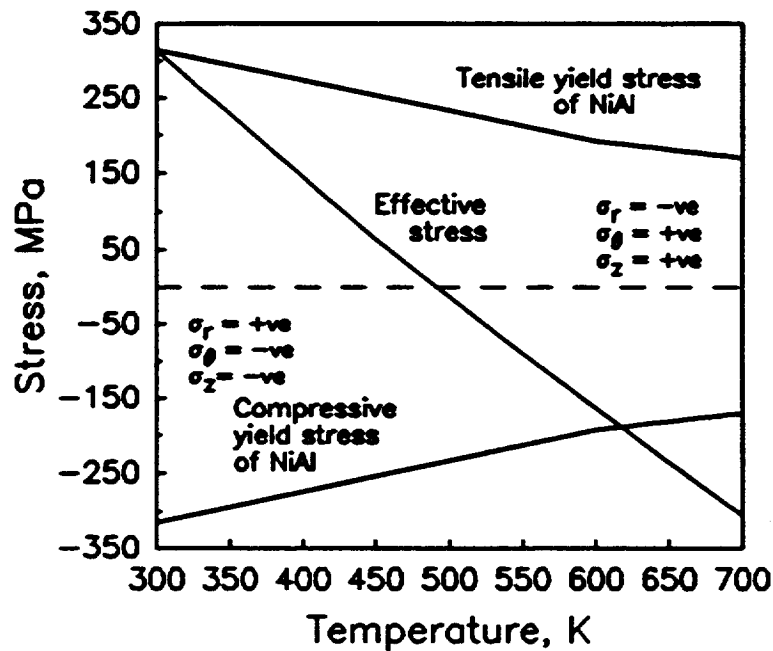
<b>Factors affecting interface bonding</b>	<b>Interface bond requirement</b>
CTE mismatch related: Radial tension during heating	Strong bond having transverse strength on the order of 75 MPa
Interface shear during cooling (near fiber ends)	Interfacial shear strength of 60-100 MPa in the temperature 600 to 1000 K
Matrix cracking stress at room temperature	Strong bond, provided multiple matrix cracking criteria is satisfied
Fracture toughness at room temperature	Weak bond ( $\Gamma_i \leq 5 \text{ J/m}^2$ ), frictional bond adequate
Load transfer at use temperature	Interfacial shear strength greater than 10-20 MPa, depending on use temperature
Ultimate tensile strength at use temperature	Interface shear strength slightly greater than 10 - 20 MPa, depending on use temperature
Creep strength at use temperature	Interface shear strength greater than 10 to 20 MPa, depending on use temperature

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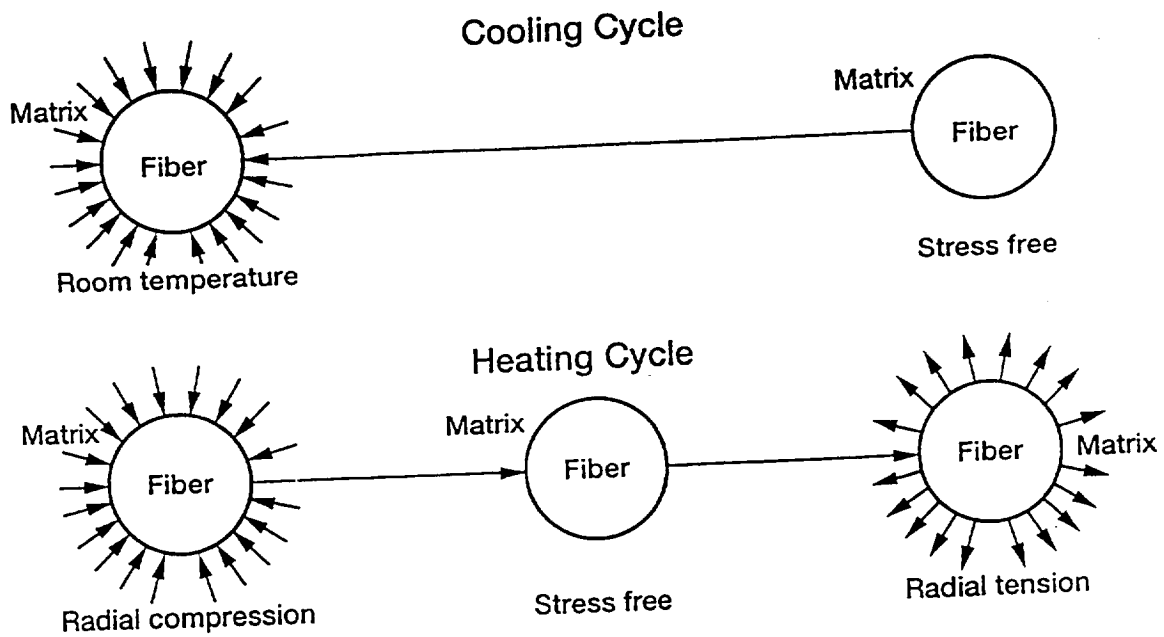
\* Various numbers quoted for interface bond strengths are based on simplistic models and should not be taken as absolute values.



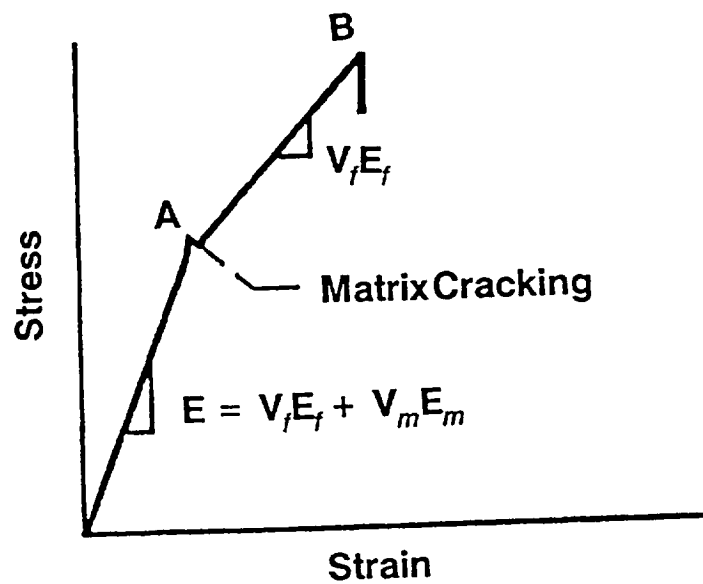
**Figure 1:** Residual effective stress ( $\sigma_{\text{eff}}$ ) in the matrix at the fiber-matrix interface during cooling of an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite as a function of temperature, along with the yield stress of NiAl. (A stress-free temperature of 1100 K is assumed)



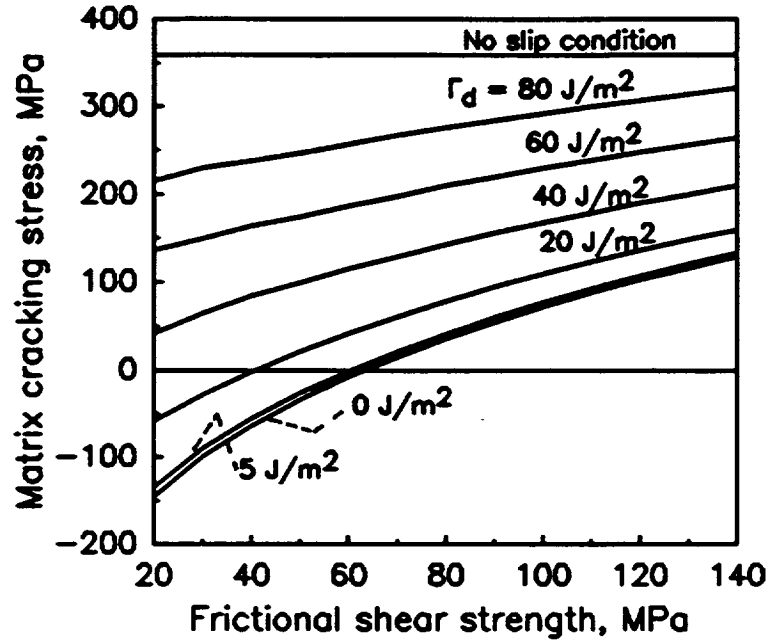
**Figure 2:** Residual effective stress ( $\sigma_{\text{eff}}$ ) in the matrix at the fiber-matrix interface during heating of an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite after the first cooldown cycle, along with the compressive and tensile yield stress of NiAl.



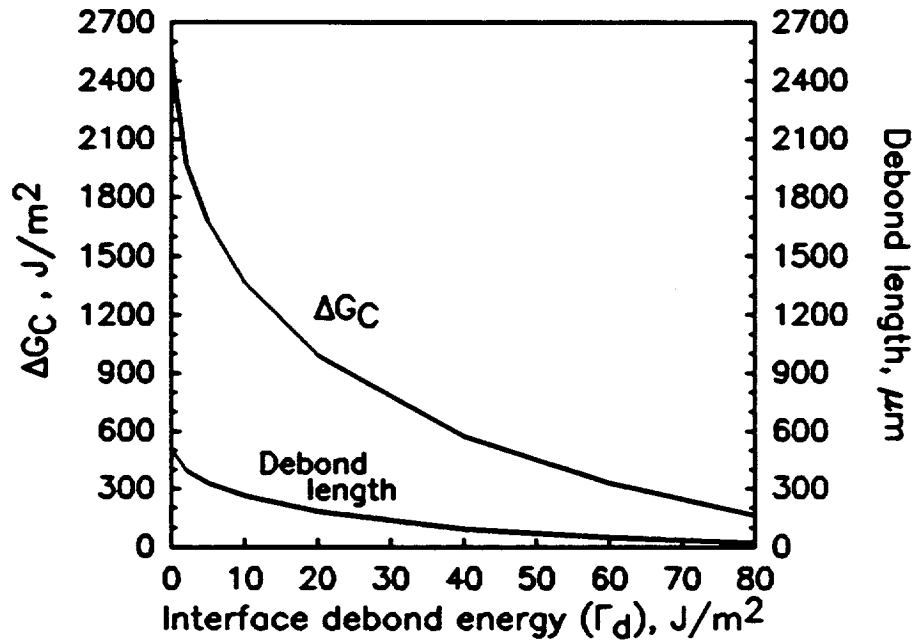
**Figure 3:** Schematic of radial stresses at the fiber-matrix interface during thermal cycling of an  $\text{Al}_2\text{O}_3/\text{NiAl}$  composite.



**Figure 4:** Schematic of the stress-strain curve for brittle matrix composites (from ref. 15).

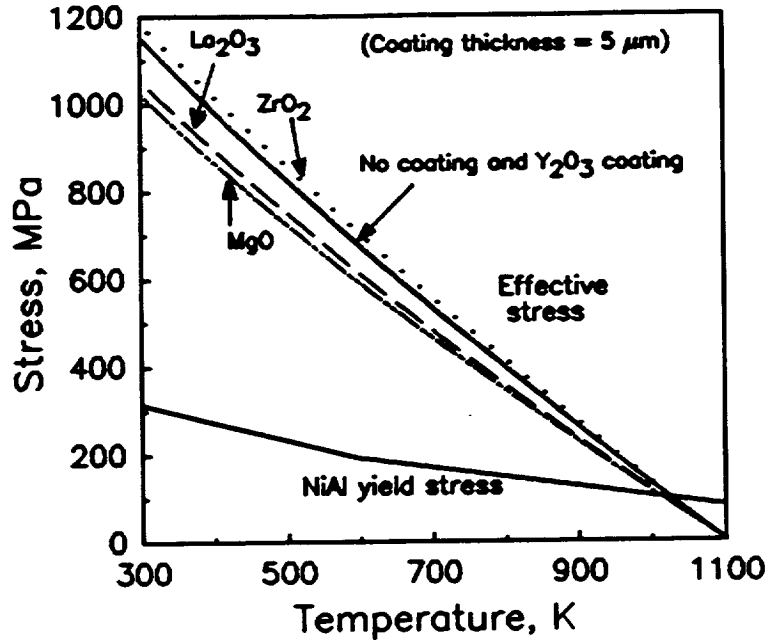


**Figure 5:** The effect of frictional shear strength at the fiber-matrix interface on steady state matrix cracking stress at room temperature for an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite. The curves are for different interface debond energy ( $\Gamma_d$ ) values.

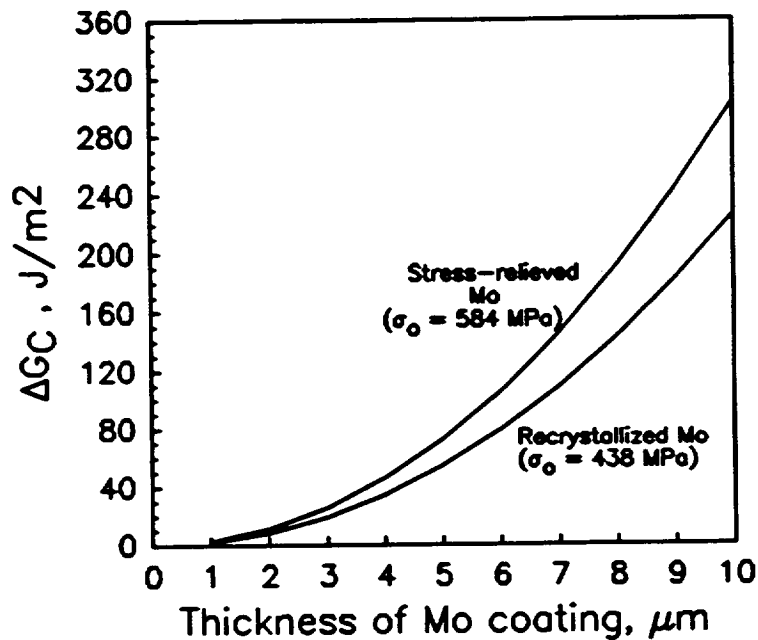


**Figure 6:** The effect of interface debond energy ( $\Gamma_d$ ) on the increase in fracture energy ( $\Delta G_c$ ) due to debonding only for an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite. The interface frictional shear strength was assumed to be 87 MPa.





**Figure 7:** The effect of different interface coatings on Von-Mises effective stress ( $\sigma_{\text{eff}}$ ) in the matrix at the matrix-coating interface during cooling of an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite as a function of temperature.



**Figure 8:** Increase in fracture energy ( $\Delta G_c$ ) for an  $\text{Al}_2\text{O}_3(30 \text{ v/o})/\text{NiAl}$  composite due to incorporation of a ductile (Mo) interface coating as a function of the coating thickness.

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